PCT WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 5 :		(11) International Publication Number: WO 93/20713
A23J 1/20, A23L 1/314	A1	(43) International Publication Date: 28 October 1993 (28.10.93)

(21) International Application Number: PCT/IE93/00021

(22) International Filing Date: 21 April 1993 (21.04.93)

(30) Priority data:
921259 22 April 1992 (22.04.92) IE

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(81) Designated States: AU, CA, JP, KR, NZ, US, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

Published
With international search report.
With amended claims.

(54) Title: PRODUCTS DERIVED FROM WHEY AND THEIR USE IN FOODSTUFFS

(57) Abstract

A process for the manufacture of a whey protein concentrate (WPC)from whey comprises the steps of reducing the pH of the whey to a pH in the range 2.5-3.5, followed by ultrafiltration. Either acid whey or sweet whey can be used as a starting material. The process can be used to manufacture WPCs with consistent specific functional properties for use in a variety of foodstuffs. If it is desired to obtain a low fat WPC or a defatted WPC, the whey is subjected to microfiltration prior to the pH reduction step. WPCs having a protein content of the order of 80-90% by weight are obtainable.

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Description

Products derived from whey and their use in foodstuffs

Technical Field

This invention relates to a process for the manufacture of whey protein concentrates (WPCs) with improved functional properties and to the use of such WPCs in the manufacture of various foodstuffs.

Background Art

WPCs are produced by subjecting whey to ultrafiltration. The WPCs obtained have a protein content in the range 35-80% by weight. WPCs with a protein content of the order of 35% by weight currently sell for ~IR£800 per tonne, whereas WPCs with a protein content of the order of 80% by weight currently sell for ~IR£4,000 per tonne.

Whey proteins, which constitute ~20% by weight of milk protein, have a high nutritional value and thus WPCs are suitable for use as, or in, foodstuffs. Whey protein has a nutritional value which is comparable to that of egg protein.

However, proteins also have functional properties which are important in the manufacture of foodstuffs, especially bakery, confectionery, dairy and meat products. These functional properties, which affect texture, include *inter alia* emulsifying, foaming, gelling and water binding properties.

Whereas one can heat whey and cause the precipitation of nutritionally valuable protein, such protein loses its functionality due to denaturation. For optimal functionality the tertiary structure of the whey protein must be maintained.

Developments in membrane filtration technology, especially ultrafiltration, have enabled one to obtain WPCs in essentially their

natural state with good functional properties. Conventionally diafiltration has been used as an adjunct to remove lactose.

To date it has not been possible to achieve a good level of consistency in the production of WPCs, which has limited their application in, and general acceptance by, the food manufacturing sector.

Also to date it has not been possible to consistently obtain WPCs with functionality equivalent to that of egg white for example, especially in terms of its gelling properties in both aqueous and saline media.

The use of ultrafiltration leads to protein enriched whey fractions. However, such protein enrichment also leads to enrichment of the fat content which has an adverse effect on the functionality, especially on foaming and gelling properties. For example, a WPC obtained by ultrafiltration with an 80% by weight protein content would typically have a fat content in excess of 8% by weight. However, the use of microfiltration before the protein extraction by ultrafiltration enables one to obtain a WPC with a low fat content. Thus it is possible to obtain a WPC with 90% protein and less than 1% fat using a combination of microfiltration and ultrafiltration. In theory such a product should have excellent properties. Such defatted WPCs show improved gelling properties in aqueous media but give similarly poor gelling performance in saline media, relative to WPCs with a normal fat content. At present low fat WPCs are used mainly for their nutritional properties.

Ion exchange chromatography, especially cation exchange chromatography, can be used to produce whey protein isolates with good functional properties, especially the ability to gel in saline media. However, the use of ion exchange chromatography is expensive and thus cannot be used to produce WPCs for use in foodstuffs at a price generally acceptable to the consumer.

Various modified WPCs are produced. Such modified WPCs are modified, for example, chemically by addition of salts such as polyphosphates and citrates, so as to give particular functional properties.

There is a need for a process which results in the production of WPCs with consistent specific functional properties for use in a variety of foodstuffs. In particular, a WPC is often sought which has a functionality comparable to that of egg white.

There is also a need for a process which can use either acid whey or sweet whey as a starting material and which results in the production of WPCs with consistently desired functionality.

Disclosure of Invention

The invention provides a process for the manufacture of a whey protein concentrate from whey, which comprises the steps of reducing the pH of the whey to a pH in the range 2.5-3.5, followed by ultrafiltration.

The process according to the invention results in the production of WPCs with consistently improved functional properties as hereinafter described and which can be incorporated in a variety of foodstuffs. Furthermore, the reduction of pH prior to ultrafiltration relative to conventional processing does not adversely affect flux rates.

Preferably, the pH of the whey is reduced to a pH in the range 2.8-3.2, more especially 3.0, before ultrafiltration.

Preferably, the pH of the whey is reduced by the addition of a food grade acid, more especially hydrochloric acid.

The starting whey may be acid whey (pH ~ 4.0-5.0) or sweet whey (pH ~ 5.8-6.8).

Acid whey is conventionally used in the manufacture of acid casein which in turn is used to produce caseinates which are typically used in the manufacture of, for example, meat products, coffee whiteners, low fat products and cheese substitutes. Sweet whey on the other hand is the normal by-product of cheese and rennet casein manufacture following the separation of the curds.

Ultrafiltration in accordance with the invention is preferably carried out using a 1,000-50,000 molecular weight cut-off (MWCO) membrane. In a particularly preferred embodiment of the invention the membrane will have a MWCO less than 15,000, especially of the order of 5,000.

Preferably, the whey is held for a period of time, suitably *circa* 1 hour, before being subjected to ultrafiltration.

The ultrafiltration may be carried out at the reduced pH or, alternatively, following upward pH adjustment to a more neutral pH, for example pH 6.3.

If it is desired to obtain a low fat WPC or defatted WPC, the whey is subjected to microfiltration prior to the pH reduction step. Preferably, the microfiltration is carried out using a microfiltration membrane with a porosity in the range 0.05-1.0 μm, suitably 0.1μm.

The product obtained in accordance with the invention, which is subjected to a combination of microfiltration and ultrafiltration, would typically have a fat content of the order of 0.5% and, therefore, it is more specifically defined as a 'defatted' WPC.

Following the ultrafiltration step, the retentate is optionally subjected to diafiltration for further removal of lactose.

Following the ultrafiltration step or the diafiltration step, as appropriate, the pH of the retentate is preferably raised to a pH in the range 6.0-7.5, followed by spray drying. The pH adjustment is preferably carried out using a food grade alkali, such as sodium

hydroxide, potassium hydroxide or calcium hydroxide. Alternatively, as indicated above, the pH adjustment may be carried out before ultrafiltration, in which case the whey product still retains the desired gelling characteristics, but has the additional advantage of a reduced mineral content, due to subsequent partial removal of the added alkali mineral during ultrafiltration/diafiltration.

It will be appreciated that drying of the product can be carried out by any suitable means, in addition to spray drying.

The invention also provides a WPC whenever manufactured by a process as hereinbefore specified.

The WPC manufactured by the process according to the invention preferably has a protein content greater than 50% by weight, more especially of the order of 80-90% by weight.

According to one aspect of the invention, there is provided a whey protein concentrate having a gel strength greater than 275g in aqueous media and a gel strength greater than 275g in 0.2M saline media when a gel containing 10% w/v protein at pH 7.0 formed after heating to 90°C for 30 min., is measured in a Stevens' LFRA Texture Analyser at a compression setting of 47%, and at a temperature of 20°C. Such a WPC has excellent functionality and thus a wide application in foodstuffs, for example in bakery, confectionery and meat products.

According to a further aspect of the invention there is provided a defatted whey protein concentrate having a gel strength greater than 525g in aqueous media when a gel containing 10% w/v protein at pH 7.0 formed after heating to 90°C for 30 min., is measured in a Stevens' LFRA Texture Analyser at a compression setting of 47%, and at a temperature of 20°C. Such products have particular application in bakery, confectionery and dairy products.

According to a still further aspect of the invention there is provided a defatted whey protein concentrate having a gel strength greater than 400g in 0.2M saline media when a gel containing 10% w/v protein at pH 7.0 formed after heating to 90°C for 30 min., is measured in a Stevens' LFRA Texture Analyser at a compression setting of 47%, and at a temperature of 20°C. Preferably, said product has a gel strength greater than 500g under the specified conditions. Such products have particular application in meat products.

Examples of meat products containing the gelling agents according to the invention are cooked meats, hamburgers, patés and sausages.

Gel strengths as hereinbefore defined are determined in accordance with a modification of a method described by Mulvihill, D.M. and Kinsella, J.E. ((1988) Journal of Food Science, *53*, No. 1, 231) as hereinafter described.

The Stevens' LFRA Texture Analyser is manufactured by Mechtric Stevens, U.K.

The invention will be further illustrated by the following Examples.

Best Modes for Carrying Out the Invention

EXAMPLE 1

2,000 l of clarified, cooled, rennet casein whey (pH 6.6, % total solids (T.S.) 6.0, temperature 6°C) was used for the production of a high gelling, defatted WPC product in the following manner.

The whey was first microfiltered to remove residual fat present in clarified whey. Microfiltration was carried out on an Alfa Laval MFS - 7 (Trade Mark) microfiltration plant, incorporating uniform transmembrane pressure (UTP) design, fitted with 0.1 μm ceramic

membranes, with an overall membrane area of 1.4 m^2. Microfiltration was operated on a continuous basis, with a permeate flux of 100 $l/m^2/h$, at a 10x concentration factor and a temperature of 50°C.. The whey was heated to 50°C. by pumping to the microfiltration plant *via* the heating section of a heat exchanger. The defatted permeate was cooled to 6°C., as it came off the microfiltration plant by pumping through the cooling section of the heat exchanger.

Approximately 1,800 l of defatted whey permeate was collected. This was heated up to 55°C., on a heat exchanger and held at this temperature for 30 min., before ultrafiltration. Also, before ultrafiltration, the pH was adjusted downward to pH 3.0, by the addition of 10% hydrochloric acid.

Ultrafiltration was carried out on a Romicon (Trade Mark) hollow fibre system (6.9 m^2 membrane area), using a modified batch mode. Ultrafiltration was carried out to a concentration factor of 40x and a 4x diafiltration step was also incorporated, resulting in a concentrate product with a protein dry matter content of greater than 85.0% by weight.

The pH of the concentrated product was adjusted upwards to pH 6.5 - pH 7.0, by the addition of 10% sodium hydroxide solution. Finally, the product was spray dried to a powdered form.

The product obtained had a protein content of greater than 85.0%, as stated, a fat content of 0.2%, and had high gel strength characteristics when tested under both aqueous and saline conditions as follows.

Preparation of aqueous gel.

A quantity of the product obtained above equivalent to 10g protein is added gradually to 60 ml., of distilled water in a 150 ml., beaker on a magnetic stirrer and stirred continuously for 30 min., or until the protein is fully dispersed. The pH of the solution is adjusted,

if necessary, to 7.0 with 0.1M HCl or 0.1M NaOH. The solution is transferred to a 100ml., volumetric flask and the contents of the beaker carefully washed out with distilled water and the solution diluted to the mark with distilled water. The contents of the flask are mixed thoroughly. The solution is then centrifuged at 500 r.p.m. for 10 min., at 20°C.. Approximately 14ml., of the solution are poured into each one of a series of prepared gelation tubes which are stoppered and clamped in a test tube rack. The rack is transferred to a water bath at 50°C., and the tubes heated for 30 min,. at 50°C.. The temperature of the water is then increased to 90°C., at a rate of 2°C., per min. and held at 90°C., for 30 min.. The rack is removed from the water bath and immersed in water at 4°C., and maintained at this temperature overnight.

The following day the rack is placed in a water bath at 20°C for 30 min., to allow the gels to equilibrate before removal from the tubes and cutting prior to tensile assessment.

Preparation of saline gel.

The above procedure is repeated except that the gels are made up using 0.2M NaCl instead of distilled water.

The gel strength or tensile assessment is carried out on a Stevens' LFRA Texture Analyser in accordance with the manufacturers' instructions and using distance of test - 3, 7 or 9 mm., for 20%, 46.7% (hereinafter 47%) and 60% compression, respectively.

In each case a 15mm height of gel is tested. Nine gels are cut for each sample to be tested - three at 20% compression, three at 47% compression and three at 60% compression. The results at 47% compression were selected as being the most representative for the products produced in accordance with the invention.

Thus for the product obtained in Example 1, the results were as follows:

Gel type	Average gel strength (g)
Aqueous gel	755
Saline gel	650

EXAMPLE 2

The procedure of Example 1 was repeated using acid whey at pH ~4.6. Average gel strengths for aqueous and saline gels were as follows:

Gel type	Average gel strength (g)
Aqueous gel	560
Saline gel	440

EXAMPLE 3

Example 1 was repeated except that the microfiltration step was omitted. The gel strengths for aqueous and saline gels prepared in accordance with the procedure of Example 1 were as follows:

Gel type	Average gel strength (g)
Aqueous gel	300
Saline gel	300

EXAMPLE 4

Example 1 was repeated except that following downward pH adjustment to pH 3.0 the product was held at 50°C. for 1 h., after which the pH was readjusted to pH 6.3 before ultrafiltration. Average gel strengths for aqueous and saline gels were as follows:

Gel type	Average gel strength (g)
Aqueous gel	570
Saline gel	250

EXAMPLE 5

Preparation of meringues

A batch of meringues was prepared using the product of Example 1 as a substitute for dried egg albumen using the following ingredients:

Ingredient	Weight (g)
Product of Example 1	12.50
Caster sugar	200.00
Water	87.50

The product of Example 1 was mixed to a paste first with an equal volume of water, followed by addition of the remaining water and mixing until full dissolution. The solution was transferred to the mixer bowl of a Hobart Kitchen Aid Food Mixer (Trade Mark) fitted with a balloon whisk attachment and whipped at speed 8 for 5 min. The sugar was then added gradually to the foamed solution over a period of 3 min.. The speed was reduced to 4-5 after half of the sugar had been added. Mixing was continued for a further 5 min., at speed 4. Using a piping bag and nozzle, the mixture was piped out on to a non-stick silicone paper into tall conical meringues. All utensils were kept completely free from fats and oils. The meringues were baked at 100°C., in a Chandley Bakery Oven (Trade Mark) or until fully baked.

Control meringues were prepared using dried egg albumen instead of the product of Example 1.

SENSORY EVALUATION RESULTS.

Characteristic	Dried Egg Albumen	Defatted WPC
Colour	Off white	Off white.
Taste	Clean	Very slight whey after taste.
Texture	Soft, sticky centre with crisp outer shell	Soft sticky centre with crisp outer shell.
Shape	Holds shape during cooking with firm peaks.	Did not hold shape and peaks sagged. However, inclusion of acid (citrate, lactate) overcame this.

Overall, the defatted WPC in accordance with the invention gave acceptable meringues at 100% replacement level of dried egg albumen.

EXAMPLE 6

Preparation of nougat

Nougat was prepared using the product of Example 1 as a substitute for egg albumen using the following ingredients:

	Ingredient	Weight (g)
A.	Product of Example 1	7.50
	Water	15.00
B.	Liquid Glucose 42DE (Trade Mark)	62.50
C.	Sucrose	380.00
	Liquid Glucose	262.50
	Water	95.00
D.	Chopped Hazelnuts	75.00
	Diced Glacé Cherries	50.00
	Icing Sugar	17.50
E.	Hydrogenated palm kernel oil (HPKO)	22.50
F.	Vanilla Flavour	0.75

Ingredients A were mixed and left aside to dissolve. B was then added to A in a Hobart (Trade Mark) mixer and beaten to a stiff foam (frappé) at high speed. Ingredients C were boiled to 94.5% solids (132°C) and added to the frappé in a thin stream, using the mixer at a lower speed. Ingredients D were added to the mixture, followed by the melted HPKO. The latter was added last to avoid foam breakdown. The vanilla flavour was added and the final mixture beaten for a further 15 sec., before depositing in a tray. The nougat was left to grain for approximately 12 h., and then cut into pieces.

Control nougat was prepared using egg albumen in place of the product of Example 1.

The nougat containing the product of Example 1 compared favourably as regards texture and flavour with the control.

EXAMPLE 7

Pork sausage product

A pork sausage product was prepared using the product of Example 1 as a partial replacer of lean meat using the following ingredients:

Ingredient	Weight (g)
Sow meat (90% lean)	16.65
Young pork meat (80% lean)	18.75
Pork fat	19.05
Rusk	14.05
Spice	2.50
Salt	1.00
Product of Example 1	5.00
Water/ice	23.00
	100.00

A combination of prechopped lean, young pork meat and sow meat and pork fat was added to a bowl chopper and chopped to uniform meat matrix. A blend of the dry ingredients (product of Example 1, spice, rusk and salt) was slowly added to the fast rotating bowl chopper. The water/ice mix was then added and the blend comminuted to a fine 'emulsion' i.e. until all the fat and water were completely absorbed.

The emulsion was then placed in a hydraulic stuffer (Mainca, Barcelona, Spain) and extruded into DEVRO (Trade Mark) dry casings and hand linked. The sausages were held overnight at 9°C., prior to evaluation *inter alia* for % cook loss, % fat loss, sensory and textural attributes relative to both unsupplemented and soya (5%) supplemented products.

The product of Example 6 and the soya supplemented product each has a partial (15%) replacement of lean meat relative to the unsupplemented product.

The results are shown in Table 1.

TABLE 1.

Comparison of product of Example 6 with unsupplemented control and soya (5%) supplemented product

Product	Emulsion*	% Cook Loss	Fat Loss	Sensory Comments
Unsupplemented Control	Good firm emulsion (score 8)	16.5	1.9	Good flavour Succulent mouthfeel.
Soya supplemented product	Very stiff over dry emulsion (score 1)	21.2	5.6	Slight after taste, rubbery texture.
Product of Example 6	Smooth emulsion slightly soft (score 5)	12.4	1.6	Very slight after taste, succulent mouthfeel.

* Emulsion scored on a scale of 1-10.

This invention is not limited to the embodiments described above which may be modified and/or varied without departing from the scope of the invention.

CLAIMS:

1. A process for the manufacture of a whey protein concentrate from whey, which comprises the steps of reducing the pH of the whey to a pH in the range 2.5-3.5, followed by ultrafiltration.

2. A process according to Claim 1, wherein the pH of the whey is reduced to a pH in the range 2.8-3.2.

3. A process according to Claim 1 or 2, wherein the pH of the whey is reduced to a pH of 3.0.

4. A process according to any preceding claim, wherein the whey is acid whey (pH ~ 4.0-5.0).

5. A process according to any one of Claims 1-3, wherein the whey is sweet whey (pH ~ 5.8-6.8).

6. A process according to any preceding claim, wherein prior to the pH reduction step, the whey is subjected to microfiltration for the removal of fat.

7. A process according to any preceding claim, wherein following the ultrafiltration step, the whey material is subjected to diafiltration.

8. A process according to any preceding claim, wherein following the ultrafiltration step or the diafiltration step, the pH of the retentate is raised to a pH in the range 6.0-7.5, followed by spray drying.

9. A whey protein concentrate whenever manufactured by a process claimed in a preceding claim.

10. A whey protein concentrate according to Claim 9, having a protein content greater than 50% by weight.

11. A whey protein concentrate according to Claim 9 or 10, having a protein content of the order of 80% by weight.

12. A whey protein concentrate having a gel strength greater than 275g in aqueous media and a gel strength greater than 275g in 0.2M saline media when a gel containing 10% w/v protein at pH 7.0 formed after heating to 90°C for 30 min., is measured in a Stevens' LFRA Texture Analyser at a compression setting of 47%, and at a temperature of 20°C.

13. A defatted whey protein concentrate having a gel strength greater than 525g in aqueous media when a gel containing 10% w/v protein at pH 7.0 formed after heating to 90°C for 30 min., is measured in a Stevens' LFRA Texture Analyser at a compression setting of 47%, and at a temperature of 20°C.

14. A defatted whey protein concentrate having a gel strength greater than 400g in 0.2M saline media when a gel containing 10% w/v protein at pH 7.0 formed after heating to 90°C for 30 min., is measured in a Stevens' LFRA Texture Analyser at a compression setting of 47%, and at a temperature of 20°C.

15. A defatted whey protein concentrate according to Claim 14 which has a gel strength greater than 500g.

16. A food product containing a whey protein concentrate according to any one of Claims 9-15.

17. A meat product containing a whey protein concentrate according to Claim 12, 14 or 15.

AMENDED CLAIMS

[received by the International Bureau on 6 September 1993 (06.09.93) ; original claim 1 amended ; other claims unchanged (1 page)]

1. A process for the manufacture from whey of a high gelling whey protein concentrate which gels in aqueous and saline media, which process comprises the steps of reducing the pH of the whey to a pH in the range 2.5-3.5, followed by ultrafiltration, the acidified whey being held for a period of at least 20 minutes prior to ultrafiltration.

2. A process according to Claim 1, wherein the pH of the whey is reduced to a pH in the range 2.8-3.2.

3. A process according to Claim 1 or 2, wherein the pH of the whey is reduced to a pH of 3.0.

4. A process according to any preceding claim, wherein the whey is acid whey (pH ~ 4.0-5.0).

5. A process according to any one of Claims 1-3, wherein the whey is sweet whey (pH ~ 5.8-6.8).

6. A process according to any preceding claim, wherein prior to the pH reduction step, the whey is subjected to microfiltration for the removal of fat.

7. A process according to any preceding claim, wherein following the ultrafiltration step, the whey material is subjected to diafiltration.

8. A process according to any preceding claim, wherein following the ultrafiltration step or the diafiltration step, the pH of the retentate is raised to a pH in the range 6.0-7.5, followed by spray drying.

9. A whey protein concentrate whenever manufactured by a process claimed in a preceding claim.

10. A whey protein concentrate according to Claim 9, having a protein content greater than 50% by weight.

INTERNATIONAL SEARCH REPORT

International Application No PCT/IE 93/00021

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)[6]

According to International Patent Classification (IPC) or to both National Classification and IPC

Int.Cl. 5 A23J1/20; A23L1/314

II. FIELDS SEARCHED

Minimum Documentation Searched[7]

Classification System	Classification Symbols
Int.Cl. 5	A23J ; A23C ; A23L

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched[8]

III. DOCUMENTS CONSIDERED TO BE RELEVANT[9]

Category °	Citation of Document, [11] with indication, where appropriate, of the relevant passages [12]	Relevant to Claim No.[13]
X Y	DE,A,2 244 999 (STAUFFER CHEMICAL CO.) 29 March 1973 see page 3, paragraph 3 - page 5, paragraph 3; claims 1,2,4-6 see page 8, paragraph 3 -paragraph 4 ---	1-5, 8-10,16 6
X	DE,A,2 155 696 (MOLKEREI J.A. MEGGLE MILCHINDUSTRIE) 7 June 1973 see page 2, paragraph 4 - page 3, paragraph 2; claims 1,2; examples 1,2 see page 4, paragraph 2 see page 5, paragraph 3 & NL,A,7 215 050 see claim 1 --- -/--	1-5, 8-10,16

° Special categories of cited documents : [10]

"A" document defining the general state of the art which is not considered to be of particular relevance

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"O" document referring to an oral disclosure, use, exhibition or other means

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"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

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IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
08 JULY 1993	22. 07. 93
International Searching Authority	**Signature of Authorized Officer**
EUROPEAN PATENT OFFICE	KANBIER D.T.

Form PCT/ISA/210 (second sheet) (January 1985)

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

Category °	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No.
X	GB,A,1 313 085 (MOLKEREI J.A. MEGGLE MILCHINDUSTRIE) 11 April 1973	1-5, 8-10,16
A	see page 2, line 118 - page 3, line 15; claims 1-3,9-14; examples 1,3,5	7
X	NETHERLANDS MILK AND DAIRY JOURNAL vol. 37, no. 1-2, 1983, MEPPEL pages 37 - 49 J.N. DE WIT ET AL 'EVALUATION OF FUNCTIONAL PROPERTIES OF WHEY PROTEIN CONCENTRATES AND WHEY PROTEIN ISOLATES, I: ISOLATION AND CHARACTERIZATION' see page 45 - page 46	1,4,5,7, 9,10
Y	DESALINATION vol. 53, 1985, AMSTERDAM pages 143 - 155 J.H. HANEMAAIJER 'MICROFILTRATION IN WHEY PROCESSING' see page 143 - page 146; figure 1	6
A	DE,A,3 212 732 (MILEI GMBH) 13 October 1983 see page 1, paragraph 2; claims 1,2 see page 3, paragraph 2 see page 6 - page 7	1,8,9, 12,16,17
A	EP,A,0 007 054 (STAUFFER CHEMICAL CO.) 23 January 1980 see page 9, paragraph 5; claim 1; example 4 see page 4, line 32 - page 5, line 15	1,16
P,A	WO,A,9 300 832 (COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION) 21 January 1993 see claims 12-14; examples 1,7,8	12-17

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO.

IE 9300021
SA 72393

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on
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